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MUZO
FLIGHT EXPERIENCE WITH THE PROGRAMMABLE MULTI ZONE FURNACE

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ABSTRACT

The MUlti ZOne (MUZO) furnace has been developed for growing Germanium (Ge) crystals under microgravity in a Get Away Special (GAS) payload. The MUZO furnace was launched with STS-47 Endeavour in September 1992. The payload worked as planned during the flight and a Ge sample was successfully processed. The experiment has given valuable scientific information. The design and functionality of the payload together with flight experience is reported in this paper.

INTRODUCTION

The MUZO furnace together with the temperature control system was originally developed for Bridgman crystal growth experiments with Ge crystals in a GAS canister. The ability to adapt the furnace to shorter microgravity times, typically the 7-14 minutes in sounding rockets, was a design criterion. Another criterion was the capability of processing samples in both gradient and isothermal modes.

The core of the furnace is a ceramic tube with 5 heaters and cooling gas channels. The control system can control all the heaters, the cooling gas flow and the Peltier pulses independently.

A successful experiment was performed with the GAS 330 that was flown on the Shuttle flight STS-47 in September, 1992. A reflight of the MUZO furnace with GAS 541 onboard STS-59 in March/April -94 is planned.

PAYLOAD DESCRIPTION

Furnace

The MUZO furnace is shown in figure 1. The furnace structure is built with two end plates, which contain the inlets for cooling gas and Peltier pulsing. The thermal expansions of the crucible and the sample are absorbed by springs in the end plates of the aluminum outer structure. The 15 thermocouples are pressed towards the sample by springs for optimal thermal contact. All structural parts are made of aluminum.

The centre of the MUZO furnace is the ceramic tube, which holds the resistive heating wires and is equipped with cooling channels which run along the sample length. The heating wires are divided in five heaters that can be controlled individually. A crucible is placed inside the tube, and the material of the crucible can be selected to be compatible with each specific sample. For growth of Ge crystals, quartz is used. The nominal sample size is 10 mm diameter and 100 mm long. Insulation between the ceramic furnace tube and the outer aluminum structure ensures reduced energy consumption as well as reduced structure temperatures. The maximum temperature in the furnace is 1300°C and the maximum heating rate is exceeding 3.5°C/s. The furnace is equipped with Peltier pulsing (25 A) at variable frequencies for tracking of the solid/liquid interface.

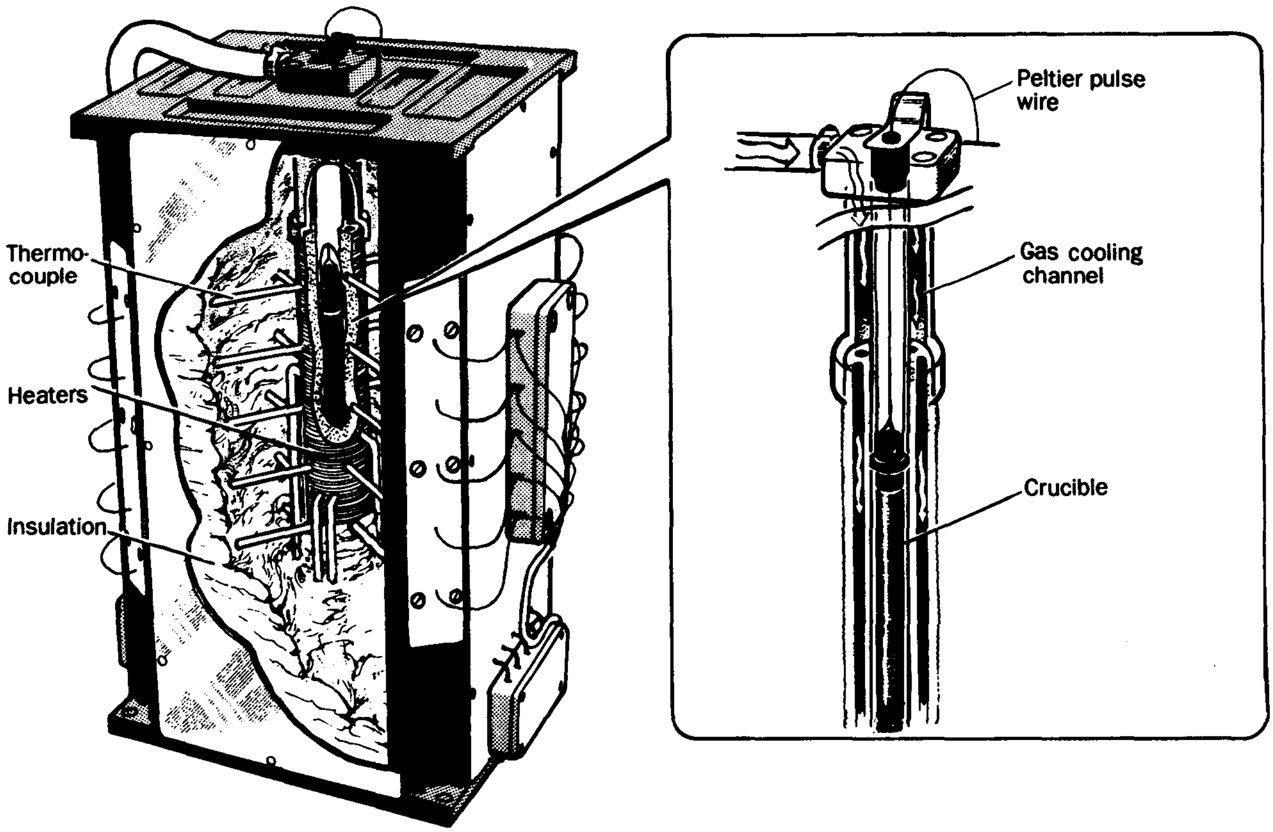

Figure 1: Cut away view of the MUZO furnace.

The design of the MUZO furnace makes it possible to change the sample in 15 minutes.

The unique feature of the MUZO furnace is the advanced temperature control system which allows for a variety of materials processing techniques ranging from isothermal to gradient growth, including rapid quench. The following growth techniques can be conceived:

- Isothermal growth
  (In-situ composite growth, High Temperature Solution Growth, Liquid Phase Epitaxy, etc.)

- Zone melting
  (Travelling Heater Method, etc.)

- Directional solidification
  (Gradient Freeze, Bridgman, etc.)

Several independently controlled heaters and a gas cooling system controlled by the furnace microprocessor, makes it possible to program:

- velocity and acceleration of solidification
- temperature gradients and profiles

<u>Power System</u>

The payload is powered with a battery unit consisting of sealed-lead batteries of standard type (which makes it a low cost design). Two sizes of cells are used (22 cells of 25 Ah, 2 V and 11 cells of 12.5 Ah, 2 V) arranged in three packages of 22 volt to optimize the total size of the power unit. A sealed compartment with venting pipes is used to accommodate the battery cells. The temperature of the batteries is monitored during the flight before the experiment starts. If the temperature is to low, a small heater that is incorporated in the battery unit is switched on to increase the temperature of the batteries to ensure maximum power delivery from the batteries. The whole battery unit holds a total capacity of 1.5 kWh.

During the vibration test of the payload some problems with the battery cells not withstanding the vibration levels occurred. The inner section of the cells containing the electrolyte moved and a fracture between the inner and outer connectors occurred. This was solved by putting three hose clamps around the cells and fixating the inner section better by pinching the cells. This rather crude method worked flawless and the battery cells capacity is still in accordance with the specification one year later.

<u>Control System</u>

The control system comprises a programmable microprocessor system. This includes thermocouple preamplifier, A/D converter, microprocessor, on board memory and power distributors for DC power to the heaters (DC power is used instead of AC to avoid the generation an oscillating magnetic field in the sample).

The software is designed for real time operation system. This makes each module independent and simplifies its adaption to a new experiment. The on board control system software is compatible with the software in the Ground Support Equipment (GSE). This facilitates the collection of data from experimental runs during testing. It also makes possible the dumping of on board memory data to the GSE. During testing, it is possible to change the experimental parameters of the control system in real time, thus decreasing the time needed to adapt to a new experiment. In addition, the GSE allows for the construction of graphs of all data collected by the system.

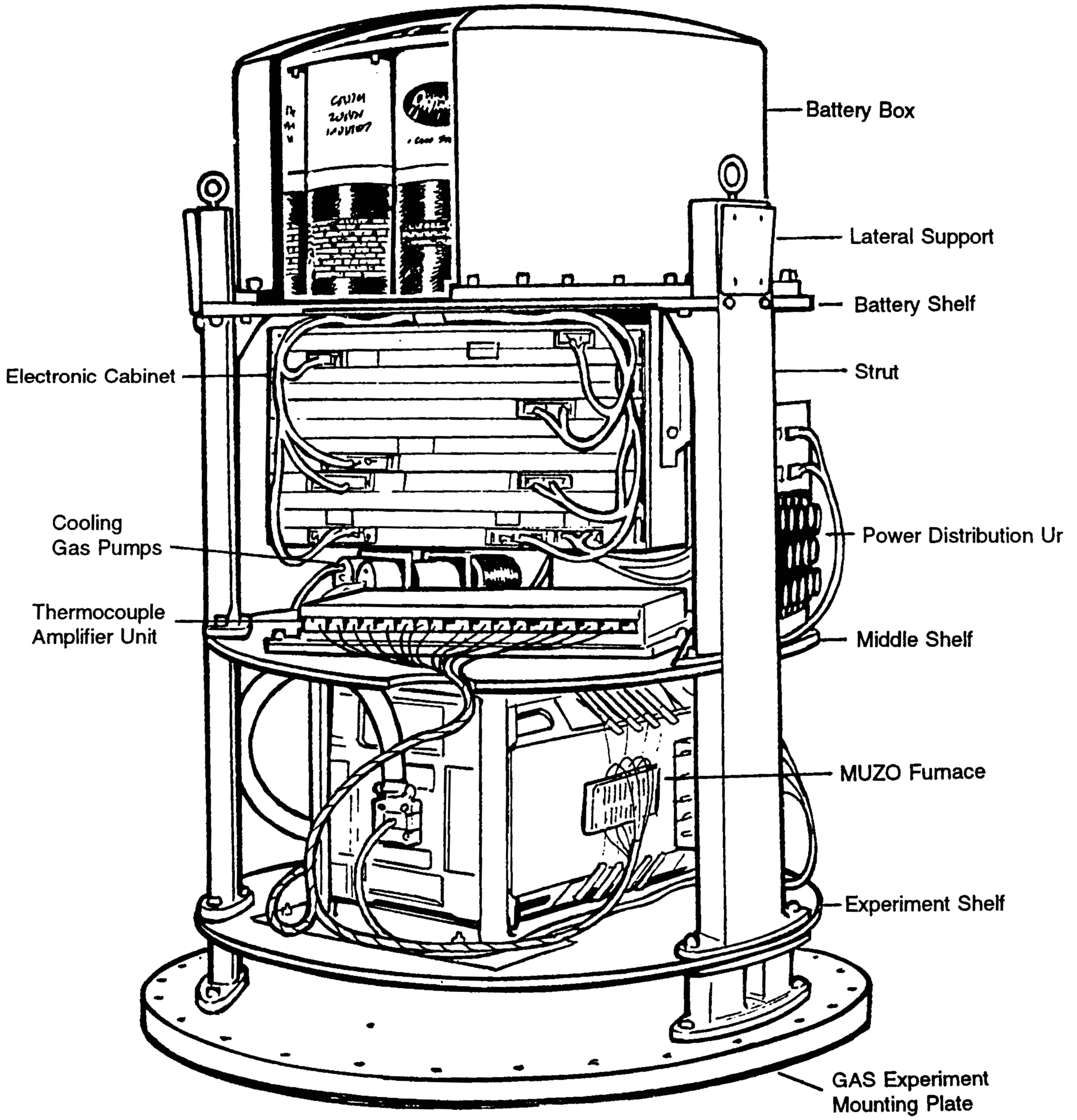

Figure 2: The GAS 330 Payload

The control system collects information from 15 thermocouples and uses the information to regulate the temperature in the sample. The control system has a software regulator for each heater. Each regulator uses different thermocouples during an experimental sequence depending upon the position of the solid/liquid interface.

During the development phase, much effort has been put into solving the problem of energy dissipation at the solid/liquid interface during solidification. Having taken the latent heat of fusion of Ge into account, the control system allows crystal growth rates and sample temperature gradients in accordance with preset values.

It is important to keep a constant temperature gradient in the molten part of the sample during the whole experiment sequence. In figure 3, temperature readings from the solidification phase of the experiment sequence are shown.

Figure 3 shows that the experiment sequence starts with a period of constant velocity (the first 10 lines from the top are separated with equal distance, each line is separated by 100 sec) and then the velocity is increasing. The sample has a melting point of 938°C. The diagram shows that the temperature gradient in the liquid part of the sample is very close to the required 2°C/mm during the whole experiment sequence.

The temperature readings in figure 3 are collected during the flight experiment. The coordinate axes are showing temperature and sample position. Each special mark in the figure indicates a position and a temperature of a thermocouple. Every 100 seconds, a line is drawn to show the temperature inside the sample, starting with the line on top.

Cooling System

The furnace core is equipped with longitudinal cooling gas channels that makes it possible to quench the sample or to achieve steep temperature gradients. The cooling system is of an open loop type and the atmosphere in the canister (argon to protect the sample from oxidation) is used as cooling medium. The gas is circulated through the furnace by two pumps. The gas flow is controlled by changing the speed of the pumps.

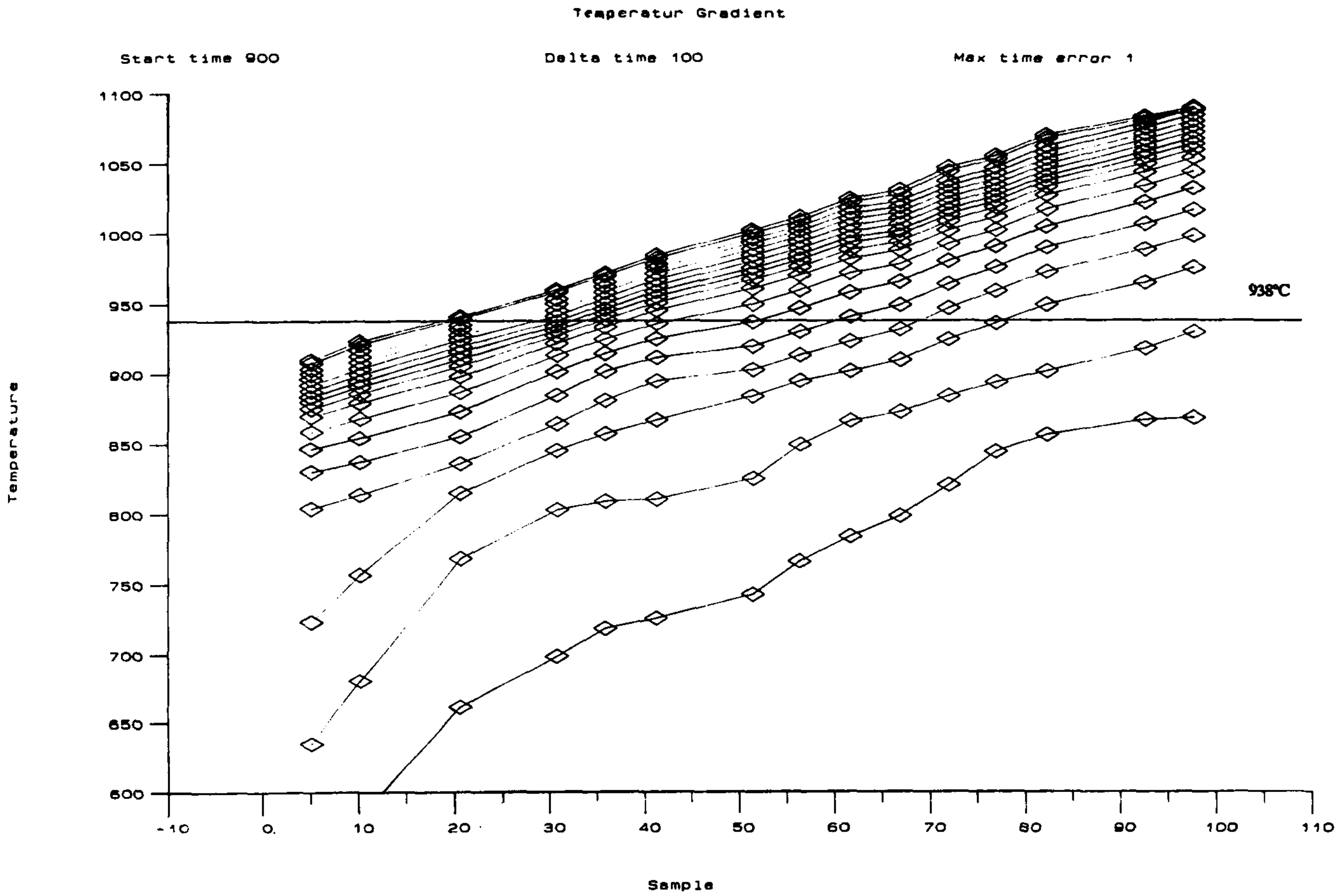

Figure 3: Axial temperature distribution in the sample at every 100 seconds, starting from top of diagram.

MICROGRAVITY EXPERIMENTS WITH Ge CRYSTALS

The first space experiments, for which the MUZO furnace has been used, was designed to study the morphological stability of a planar solid/liquid interface. Accurate control of temperature gradients and growth rates are therefore necessary. A growth rate profile starting with a period of constant rate, to obtain steady state conditions, and followed by a period with acceleration have been chosen.

The growth rate could be followed by interface demarcations obtained by Peltier pulsing every third second. The demarcations also revealed the interface shape, which was slightly concave, i.e. the centre growing about 0.5 mm behind the periphery. Of course the development of the morphological instability can also be followed by the demarcations.

A way to characterize the furnace performance, in terms of convection level in the melt, is to study axial concentration profiles. This has been done in 10 mm diameter Ge crystals doped with Ga. Results from 9 crystals grown under different convection conditions are shown in figure 4. The growth rate profiles are for all samples identical. The space sample shows a segregation typical for totally diffusion controlled growth, i.e. negligible convection. The crystal, grown in a vertical, thermally stabilizing condition, shows a segregation behaviour typical for mixed convective-diffusive transport. At about 13 mm the concentration starts to increase as a result of increased growth rate. At the same position in the space sample the initial transient was not ready and steady state was never reached.

As a summary of the tests it can be concluded that the furnace is well suited for studies of convective effects on segregation, on ground as well as in space.

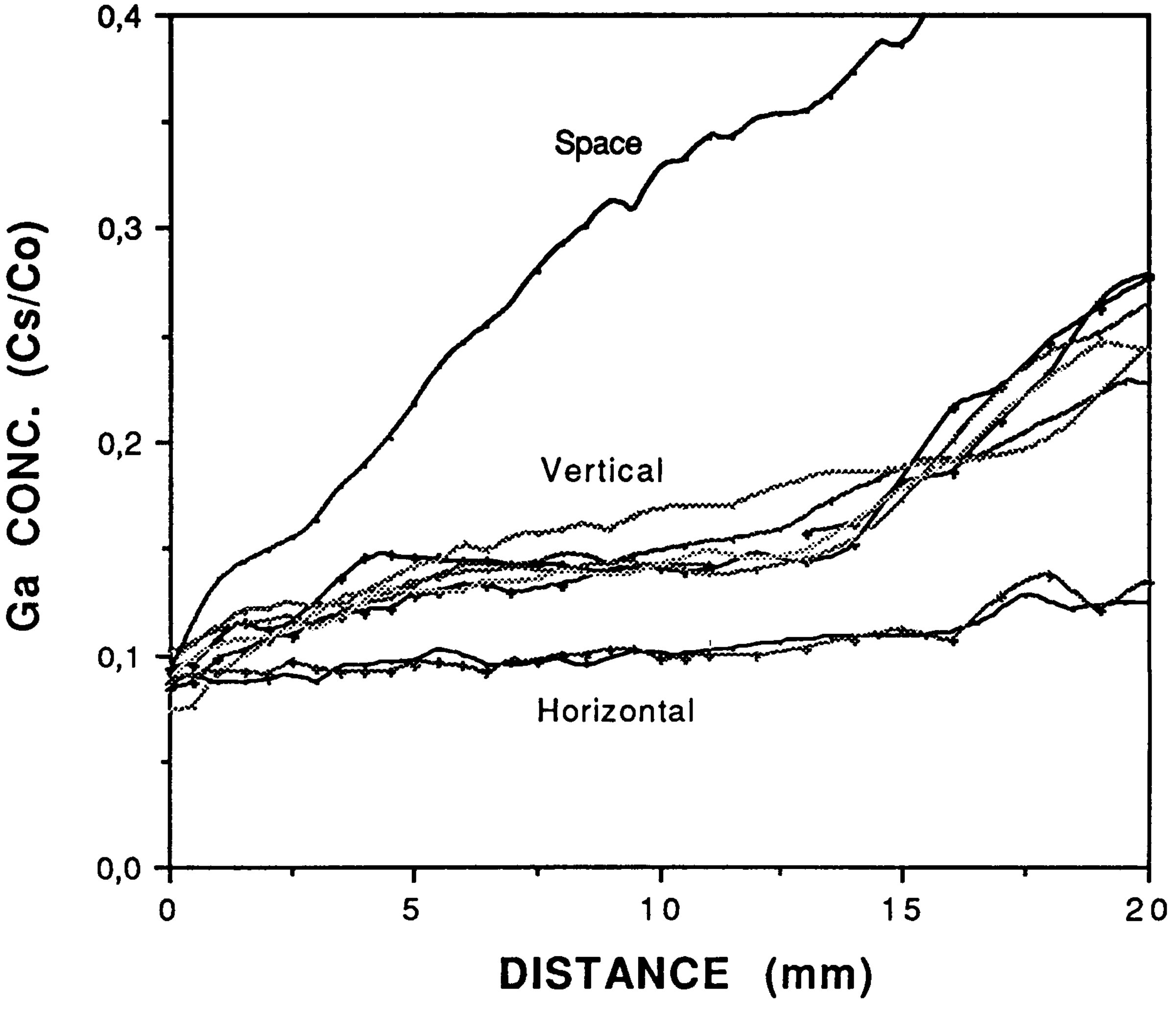

Figure 4: Normalized concentration profiles from the centre line of Ge crystals grown under different convection conditions. One crystal was grown in space. Six crystals were grown on ground in vertical, thermally stabilized with hot end up, position. The remaining two crystals were grown in a horizontal position.

ALTERNATIVE EXPERIMENT APPLICATIONS

Liquid Phase Epitaxy

The Liquid Phase Epitaxy (LPE) technique is a method of significant importance for the growth of advanced semiconductors. Microgravity experiments with the LPE technique is of major scientific and technological interest. However, the LPE technique poses severe thermal requirements on the experiment equipment and has rarely been applied in microgravity.

The thermal requirements of the LPE technique should be fulfilled by the MUZO furnace which is designed, inter alia, for high-temperature isothermal applications such as high temperature solution growth.

In-Situ Produced Composite Material

Metal Matrix Composite (MMC) is used in many applications today and one such is carbides bounded by a metal matrix. This material can be produced by in-situ precipitate carbides in a metallic liquid. This could be done by letting two materials with different composition react with each other. A precipitation of small crystals can occur at this reaction, which will give the composite.

The reaction between the two materials in liquid form often give rise to concentration and temperature gradients. The caused convection in the melt will influence the precipitation process.

In-situ produced composite material experiments requires an isothermal furnace for medium to high temperature applications. This should be fulfilled by the MUZO furnace.

CONCLUSIONS AND RESULTS

The MUZO furnace has so far demonstrated very interesting results during an experiment under microgravity conditions and will therefore be utilized for a new experiment in a GAS canister in 1994.

The batteries that were reinforced due to problems during the vibration tests functioned perfectly during flight and there has been no reduction in battery capacity one year after the flight.

The maximum temperature in the payload during flight due to internal and external heating has been between 40-50°C depending on the location inside the payload.

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